# Pliocene vertebrate locality of Çalta, Ankara, Turkey. 11. Isotopic investigation

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#### ABSTRACT

Carbon and oxygen stable isotopic abundances of fossil mammals can provide valuable palaeoenvironmental information, provided that diagenesis did not alter the biogenic signal beyond recognition. An isotopic investigation of mammal bones and teeth from Çalta (Pliocene, Turkey) demonstrates that enamel can be used for palaeoenvironmental reconstruction, but that a diagenetic signal has been overprinted on dentine and bone. By comparison with the results of similar studies on the Miocene Turkish sites of Pasalar and Kemiklitepe, it appears that the environment of Çalta was open and probably steppic, a conclusion in agreement with the study of rodent faunas.

KEY WORDS
C-13,
carbonate apatite,
mammals,
O-18,
paleodier,
Pliocene,
Turkey,

#### RÉSUMÉ

Le gisement de vertébrés pliocènes de Çalta, Ankara, Turquie. 11. Investigation isotopique. Les abondances isoropiques en carbone et en oxygène des os et des dents de mammifères fossiles peuvent apporter des informations concernant les paléoenvironnements, à condition que la diagenèse n'ait pas altéré le signal biogénique. Une étude isotopique d'os et de dents de mammifères de Çalta (Pliocène, Turquie) montre que l'émail peut être utilisé pour les reconstitutions paléoenvironnementales, mais qu'un signal diagénétique s'est surimposé sur la dentine et l'os. Par comparaison avec les résultats d'études similaires menées sur les sites du Miocène de Turquie de Pasalar et Kemiklitepe, il apparaît que l'environnement de Çalta était ouvert et probablement steppique, une conclusion en accord avec les résultats de l'étude des faunes de rongeurs du site.

MOTS CLÉS C-13, carbonate apatite, mammifères, O-18, paléodière, Pliocène, Turquie.

#### INTRODUCTION

Palaeoenvironmental reconstruction is one of the goals of the study of fossil vertehrates. During their lifetime, vertebrates record in their tissues geochemical data linked to their environment. One case particularly televant for vertebrate palaeontology is the fact that the carbonate ions incorporated within the apatitic lattice of hone and tooth minerals are in isotopic equilibrium with the bicarbonate in circulating blood serum. Thus, the isotopic signatures of carbon and oxygen of these ions are linked to dictary and environmental parameters at the time of their incorporation, such as the kind of plants at the beginning of the food web and the origin of drinking water (Krueger & Sullivan 1984; Lee-Thorp 1989; Bocherens & Mariotti 1992; Iacumin et al. 1996). Provided that these isotopic signatures have not been altered during diagenctical processes, they represent potential tools that can be used to decipher dietary habits and life environments of fossil specimens. Some examples of studies using this approach are the determination of diet differences in two Pleistocene baboon species (Lee-Thorp et al. 1989), and the changes in vegetation cover during the Cenozoic in Pakistan (Quade et al. 1992) and in South America (MacFadden et al. 1994). By using isotopic variations recorded in different teeth from the same fossil individuals in some favorable cases, it is even possible to get information on dietary and/or environmental changes that occurred during the individual lifetime (Koch et al. 1989).

Isotopic investigations have already been performed in Turkish Genozoic faunas, i.e. the middle Miocene fauna of Pasalar (Quade et al. 1995) and the late Miocene fauna of Kemiklitepe (Bocherens et al. 1994b). In this context, it was interesting to perform an isotopic study of the Pliocene fauna of Çalta.

#### MATERIAL AND METHODS

The fossiliferous locality is close to the village of Çalta, around sixty kilometers northwest of Ankara, in central Turkey. A first mention about

this site is to be found in Ozansoy (1955). The site has yielded a rich vertebrate fauna in the 1970's (Ginsburg et al. 1974; Sen et al. 1974; Sen 1977, this volume). The age of the locality is Pliocene (Sen 1977).

Tooth dentine and enamel, bone and sediment samples have been analyzed isotopically, including specimens belonging to species with herbivorous (Giraffa, bovid and Hipparion heintzi) and carnivorous (a hyenid, Chasmaportetes and a racoon-dog, Nyctereutes) habits. The choice of these two kinds of specimens have been dictated by the report of differences in the carbon isotopic abundances in herbivores and carnivores from temperate and cold areas (Bocherens & Mariotti 1992), although such differences have not been observed in South Africa (Sillen & Lee-Thorp 1994). Isotopic abundances of enamel, and of bone and dentine, have been compared in order to check for diagenetic alteration since dentine and bone are much less stable to diagenetic alteration than enamel (Lee-Thorp & van der Merwe 1987; Koch et al. 1990)

Preparation of bone and tooth cathonate hydroxylapatite has been performed according to the protocol used by Lee-Thorp (1989), modified according to Bocherens et al. (1991). All samples were cleaned and enamel was separated from dentine using a dentist wheel. The powdered teeth were treated with 1 M acetic acid-Ca acetate buffer for twenty-four houts to leach diagenetic carbonate minerals, and then rinsed with distilled water several times. They were reacted with 100% phosphoric acid at 50 °C for twelve hours. The evolved carbon dioxide was purified by cryogenic distillation in a vacuum line and introduced in a VG SIRA 9 gas source isotopic ratio mass spectrometer for measurement of its carbon and oxygen isotope compositions. Carbon dioxide was extracted from the sediment samples the same way. Isotopic abundances are normalized to international laboratory calcite standards analyzed concurrently with the aparite samples. The delta value for each isotope is calculated as  $\delta^{E}X = [(R_{sample} / R_{sample}) - 1] \times 1000$ , where  $\delta^{L}X = \delta^{Lj}C$  or  $\delta^{18}O$ , and  $R = {}^{13}C/{}^{12}C$  or <sup>18</sup>O/<sup>16</sup>O, respectively. The standards are PDB for carbon and SMOW for oxygen. Analytical precision was better than 0.1% for δ13C and 0.2%

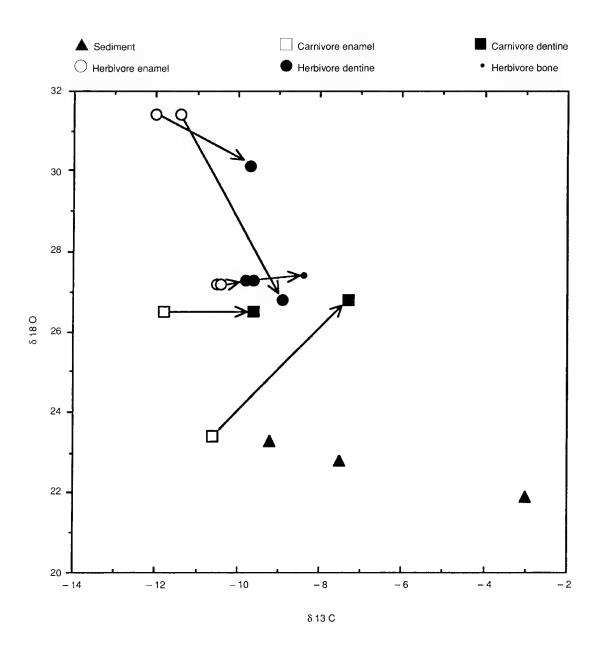


Fig. 1. — Variations of carbonate hydroxylapatite  $\delta^{13}$ C and  $\delta^{18}$ O values between different mineralized tissues of single individuals and species in Çalta.

for  $\delta^{18}$ O values. Since the correction formula is not known for oxygen in carbonate hydroxylapatite, the correction formula for calcite at the same temperature was used (Koch *et al.* 1989).

## **RESULTS**

The results of isotopic analyses are presented in table 1. In the sediment, the  $\delta^{13}C$  and the  $\delta^{18}O$ 

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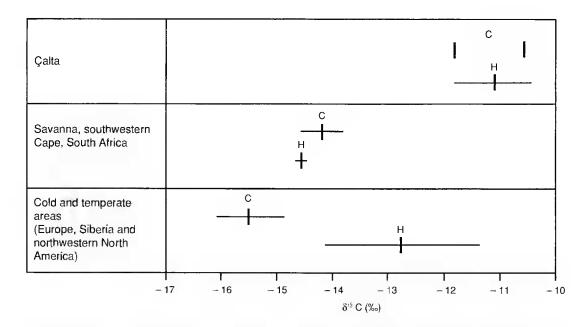


Fig. 2. — Variations of carbonate hydroxylapatite  $\delta^{13}$ C values according to trophic level in Çalta (enamel), modern South African savanna (Sillen & Lee-Thorp 1994) and modern cold and temperate areas (Bocherens & Mariotti 1992).

values range from -9.2 to -3.0% and from 21.9 to 23.3% respectively. The  $\delta^{13}$ C value of *Hipparion* bone is -8.4%,  $\delta^{13}$ C values in dentine range from -9.8 to -8.9% in herbivores and from -7.3% to -9.6% in carnivores, whereas  $\delta^{18}$ O value is 27.4% in *Hipparion* bone, and  $\delta^{18}$ O values range from 26.5 to 30.1% in dentine and from 23.4 to 31.4% in enamel.

#### DISCUSSION

An ecological interpretation of the measured isotopic abundances is possible only if the biogenic values have not been significantly altered by diagenetical processes. Thus the first part of the discussion will deal with diagenetic aspects of the results, whereas the second part will discuss possible implications.

Preservation of biogenic isotopic abundances

Two approaches can be used to estimate the degree of possible alteration of the isotopic abundances: the isotopic differences between altered (bone, dentine) and possibly non altered tissues

(enamel), and the disruption of biogenic isotopic signals, such as the difference between herbivores and carnivores.

Plotting the differences in the  $\delta^{13}$ C and the  $\delta^{18}$ O values of bone, dentine and enamel in the same specimens for the different analyzed species clearly shows that dentine  $\delta^{13}$ C values are systematically less negative than those of enamel of the same specimens, whereas  $\delta^{18}$ O values rend to be less scattered for dentine than for enamel (Fig. 1). Since the sediment  $\delta^{13}$ C values are less negative than those of all enamel samples, an alteration of the biogenic carbon isotopic composition would lead to an increase of the resulting  $\delta^{13}$ C values, which is the case for the  $\delta^{1,3}$ C values measured in dentine when compared to those measured in enamel of the same teeth, and this increase is even more important in the case of Hipparion bone, relative to dentine. In the case of oxygen, the  $\delta^{18}$ O values of sediment are lower than those of enamel. However, not all dentine δ<sup>t8</sup>O values appear lower than those of the enamel of the same tooth. Some are roughly equal (Hipparion and hyena), and one is clearly higher (racoondog). It looks as if the dentine  $\delta^{18}$ O values tend to reach an average  $\delta^{18}$ O value close to 26‰,

TABLE 1. —  $\delta^{13}$ C and  $\delta^{18}$ O values of samples from Çalta. Due to the small size of the teeth, three specimens have been pooled together in the case of *Nyctereutes*. Enamel values presented in bold have been used for figure 3.

Sample	ple Taxon Piece Number		Number	δ <sup>13</sup> C (‰)		
Sediment Carbonated concretion			100500 100600	- 9.2 - 7.5	23.3 22.8	
from palaeosoil Carbonated			100700	- 3.0	21.9	
hymenoptere nest						
Giraffe	Giraffa sp.	enamel	100800	<b>– 12.0</b>	31.4	
		dentine	100800	-9.7	30.1	
Bovid	Bovidae indet.	enamel	100900	<b>– 11.4</b>	31.4	
		dentine	100900	-8.9	26.8	
Hyena	Chasmaportetes sp.	enamel	101000	<b>- 11.8</b>	<b>26.5</b>	
		dentine	101000	-9.6	26.5	
Racoon-dog	Nyctereutes sp.	enamel (3)	101100	- 10.6	23.4	
	· · · · · · · · · · · · · · · · · · ·	dentine (3)	101100	-7.3	26.8	
Hipparion	Hipparion heintzi	enamel	103400 (ACA-255)	- 10.4	27.2	
		dentine	103400 (ACA-255)	- 9.6	27.3	
Hipparion	Hipparion heintzi	enamel	103500 (ACA-266)	- 10.5	27.2	
	i iippairoii iioiiitzi	dentine	103500 (ACA-266)	- 9.8	27.3	
Hipparion	Hipparion heintzi	bone	103600 (ACA-98)	- 8.4	27.4	

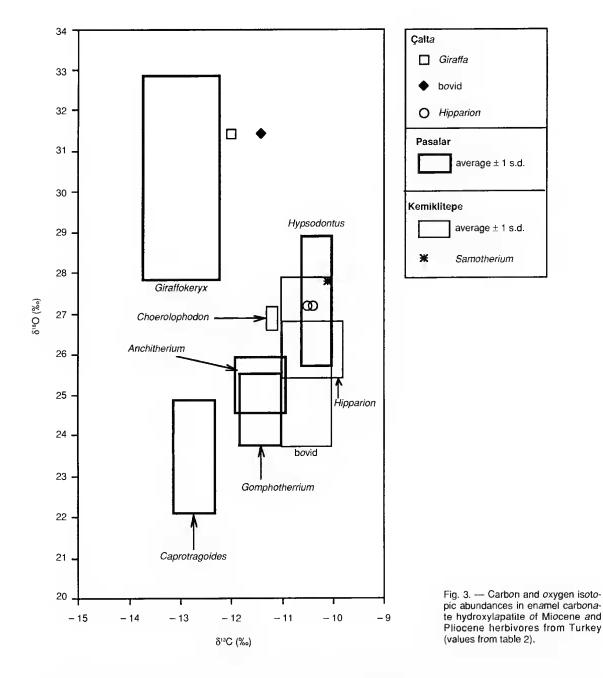
around 3‰ higher than the value of sediment carbonate. It appears thus that only enamel isotopic composition can eventually be used for palaeobiological reconstruction, in agreement with previous studies (Wang & Cerling 1994).

The  $\delta^{13}$ C values of herbivore and carnivore enamel do not appear different, which is similar to what is observed in modern South Africa (Sillen & Lec-Thorp 1994) but different from what is observed in modern and Pleistocene Europe (Bocherens & Mariotti 1992; Bocherens et al. 1994a), However, the absolute values are less negative in the Calra samples than in the modern South African ones. This could be due to the depletion in <sup>13</sup>C recorded in modern atmospheric CO, that result from the industrial development since 150 years (Friedli et al. 1986), as suggested by Lee-Thorp (submitted). This last author considers that modern values should be shifted by 1.5‰ (less negative) before comparisons are made with 813C values measured on fossils. After such a correction, the Calta values still appear less negative than the South African ones, but the difference is not very large. Moreover, the δ<sup>13</sup>C values measured on Çalta samples appear just slightly higher than those of modern herbivores from temperate and arctic environments,

and very similar if a 1.5‰ increase is considered for modern values. Thus, the δ<sup>13</sup>C values measured on Çalta samples do not appear contradictory with data gathered from modern environments.

As far as enamel  $\delta^{18}$ O values are concerned, the comparison with modern biogenic signals is not as straightforward as for carbon. A clear difference was demonstrated for hippopotamuses relative to terrestrial herbivores, in modern and fossil environments (Bocherens et al. 1996). Unfortunately, no hippopotamus has been found at Calta. Another isotopic particularity found in modern herbivores is higher δ18O values in giraffes relative to other herbivores, probably due to their dietary and drinking habits (Quade et al. 1995). It is noreworthy that among Çalta herbivores, the giraffe specimen present the highest  $\delta^{18}$ O value (Table 1). Finally, lower  $\delta^{18}$ O values have been reported for carnivotes relative to herbivores among modern mammals from Kenya (Ambrose 1992). Although the number of samples is low, both Calta carnivores present lower δ<sup>18</sup>O values than any analyzed herbivore from the site (Table 1).

Since no expected biogenic signal indicate significant alteration of *in vivo* isotopic signatures, it



is legitimate to assess some palaeobiological implications of these isotopic compositions, which will be done in the next section.

PALAEOBIOLOGICAL IMPLICATIONS OF ISOTOPIC SIGNATURES

The carbon isotopic compositions of all the specimens indicate that no C4 grasses were present

in their dietary components. This situation is similar to modern central Anatolia, which belongs to the Euro-Siberian phytogeographic region (Klein 1994). It is noteworthy that collagen carbon isotopic abundances in modern herbivores from Turkey showed no evidence of C4 grasses consumption either (Bocherens 1992).

The isotopic differences between herbivorous

TABLE 2. — Average values and standard-deviations of  $\delta^{13}$ C and  $\dot{\delta}^{18}$ O values of terrestrial herbivore enamel from Pasalar and Kemiklitepe used for figure 3. Pasalar data are from Quade *et al.* (1995) and Kemiklitepe values are from Bocherens *et al.* (1994b).

Site	Taxon	group	n	δ <sup>13</sup> C (‰)	δ <sup>18</sup> Ο (‰)	
Pasalar	Hypsodontus pronaticornis	bovid	5	$-10.3 \pm 0.3$	27,3 ± 1.6	
Pasalar	Gomphotherium pasalarense	mastodont	4	$-11.4 \pm 0.4$	$24.6 \pm 0.9$	
Pasalar	Giraffokeryx aff. punjabiensus	giraffe	4	$-12.7 \pm 0.5$	$30.3 \pm 2.5$	
Pasalar	Anchithenum aurelianense	equid	3	$-11.4 \pm 0.5$	$25.2 \pm 0.7$	
Pasalar	Caprotragoides stehlini	bovid	4	$-12.7 \pm 0.4$	$23.4 \pm 1.4$	
Kemiklitepe	Choerolophodan sp	mastodont	3	$-11.2 \pm 0.1$	$26.9 \pm 0.3$	
Kemiklitepe	Hipparion sp.	equid	4	$-10.4 \pm 0.6$	$26.1 \pm 0.7$	
Kemiklitepe	Samotherium sp.	giraffe	1	-10.1	27.8	
Kemiklitepe	boyld sp.	bovid	3	$-10.5 \pm 0.5$	25.8 ± 2.1	

taxa can provide palaeoenvironmental information. The isotopic investigation of the rich Pasalar fauna has shown that terrestrial herbivorous cluster according to their taxonomic affinities (Quade et al. 1995). It seems that a tendency can be drawn from low δ<sup>13</sup>C and δ<sup>18</sup>O values to higher  $\delta^{13}$ C and  $\delta^{18}$ O values (Table 2, Fig. 3). This tendency could reflect the fact that plants with low δ13C values are those from closedcanopy forests, where high humidity maintains low δ<sup>18</sup>O values in herbivores whereas plants with higher δ<sup>13</sup>C values are characteristic of open environments, where more evaporation in plants lead to increased 818O values in leaf water, and consequently in herbivores feeding on such plant material (Quade et al. 1995). One notable exception to this pattern is the case of browsing giraffes, with low  $\delta^{11}$ C values and high  $\delta^{18}$ O values, such as Giraffokeryx from Pasalar and Giraffa from Çalta (Fig. 3). This singular pattern can nonetheless be explained by high evaporation in top canopy leaves eaten by giraffes (Quade et al. 1995). It is noteworthy that the giraffid Samotherium does not fit into this pattern, but this genus has been recognized as less specialized in browsing than typical giraffes (Solounias et al. 1988). The isotopic composition of the different taxa of Calta herbivores fit to this pattern defined from Pasalar and Kemiklitepe studies, and more particularly to the most open environmental pole of the pattern (Fig. 3). The conclusion based on isotopic biogeochemistry of an open environment in Calta is in agreement with the conclusions based on the high proportion of rodents adapted to steppic and open environ-

ments, such as *Pseudomeriones* and *Pliospalax* and on the low proportion of murids (Sen 1977), as well as on the low number of Soricidae species (Reumer this volume). It is interesting to notice that the analyzed hipparions belong to the robust species H. heintzi, described as a subunguligrade walking on a soft soil in an arid context rather than to humid conditions (Eisenmann & Sondaar this volume). The isotopic results confirm that the analyzed individuals experienced an open environment with arid conditions. The occurrence of the Suidae Sus arvernensis in Calta is interpreted as reflecting a forested environment (Guérin et al. this volume). Unfortunately, it was not possible to perform isotopic measurements on enamel from this species, which could have allowed to verify that the suid specimens from Calta actually lived in a forested environmenr.

It is difficult to compare these isotopic abundances in terms of global climatic changes from the lower Miocene to the Pliocene because of the large variations of  $\delta^{18}$ O values between different samples from a same site. The micro-environmental factors seem to have a large influence on the oxygen isotopic abundances of fossil mammals. In order to prevent this phenomenon to bias the climatic record, it should be necessary to select a given taxon with well-known relationships between the  $\delta^{16}$ O values in meteoric waters and those of enamel apatite. An additional complicating factor is the necessity to work on enamel due to the poor stability of bone during fossilization. Unfortunately, enamel presents significant isotopic variations from one tooth to

the other, or even within one tooth, due to the fast mineralization of this tissue, which records environmental parameters during a brief instant of the individual lifetime (Bryant et al. 1996; Lee-Thorp et al. 1997). However, this problem can be turned into an advantage in the case of studying short-term climatic variations, such as seasonal changes.

## CONCLUSIONS

The isotopic biogeochemistry of carbonate hydroxylapatite of Pliocene mammals from Çalta shows that only enamel can be used for palaeoenvironmental reconstructions, and that herbivorous taxa indicate an open environment, with a rather important evapotranspiration in plants. These conclusions are in total agreement with those based on the study of the rodent fauna.

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